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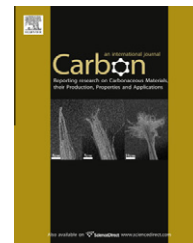
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Effectiveness of sorting single-walled carbon nanotubes by diameter using polyfluorene derivatives

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ABSTRACT

Semiconducting single-walled carbon nanotubes (SWCNTs) sorted by conjugated polymers are of great interest for electronic and optoelectronic applications. Here we demonstrate by optical methods that the selectivity of conjugated polymers for semiconducting SWCNTs is influenced by the structure of their side-chains and/or the molecular weight of the macromolecules, and that side chain functionalities determine the solubility in different dispersion media. Moreover, high resolution time-resolved photoluminescence measurements provide evidence of energy transfer from tubes with larger band gaps compared to those with smaller band gaps coexisting in SWCNT bundles.

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1. Introduction

SWCNTs are hollow cylinders composed only of carbon atoms that have remained at the forefront of nanotechnology research for the last two decades [1–3]. Because of the strong tendency of carbon nanotubes to aggregate and to form bundles, the first few studies on the optical properties of SWCNTs had limited success in explaining the photophysics of these one-dimensional objects. Since the first result of band gap photoluminescence from SWCNTs dispersed with sodium dodecyl sulfonate (SDS) in aqueous solution was reported [4], many molecules and macromolecules, including sodium dodecylbenzene sulfonate (SDBS) [5], sodium cholate, sodium deoxycholate [6,7], and single-stranded DNA, [8] have been used for the same purpose. Other methods for selective separation of SWCNTs include electrophoresis, chromatography, and density gradient ultracentrifugation [9–11].

Recently, π -conjugated polymers have proven to be very effective in solubilizing specific SWCNTs. Among these polymers, polyfluorene homo- and co-polymers show unique selectivity for a narrow distribution of semiconducting species when dissolved in organic solvents [12]. The photophysical properties of such polymer/SWCNT hybrids were explored

from many aspects at later stage. Recently, Hwang et al. reported on the polymer structure and solvent effects on the selectivity of SWCNTs dispersion [13]. Other authors have studied the interaction between the wrapped polymer chains and SWCNTs [14,15]. However, the mechanism of the unique interaction of poly(9,9-di-*n*-octylfluorenyl-2,7-diyl) (PFO) and its co-polymer with SWCNTs is still a matter for investigation. The fact that many of the co-polymers used have had different backbone structures compared to that of PFO serves to complicate relative studies. Moreover, water-soluble polyfluorene derivatives may also evidence selectivity for SWCNT dispersion, which might be helpful for the application of SWCNTs in biology. However, up to now no such studies have been reported. It would therefore be highly desirable to gain a deeper understanding of the role of the backbone and side-chain structures on the selectivity for specific SWCNT.

In our work, we studied the photophysics of SWCNT dispersion with amine-functionalized polyfluorene (poly(9,9-di-(*N,N*-dimethylaminopropyl)fluorenyl-2,7-diyl) (PFDMA)) and its ammonium salts [(*N,N,N*-trimethylammonium)-propyl]-2,7-fluorene dibromide (PFAB) in different media. We found that the selectivity for SWCNTs of PFDMA in toluene is different than that of PFO.

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New absorption and photoluminescence (PL) peaks appear in PFDMA/SWCNTs spectra, indicating the presence of a larger number of nanotube species. SWCNT dispersions with PFAB in D₂O contain similar species to the PFDMA-based dispersion in toluene, with a universal shift of the PL peaks due to the change of effective dielectric constant of the medium. Photoluminescence decays of dispersed SWCNTs indicate that the intrinsic lifetime of the 'bright' exciton in SWCNTs is in the range of 28–40 ps. More importantly, the PL lifetimes of carbon nanotubes with different band-gaps show diverse lifetimes, indicating exciton energy transfer in residual SWCNT bundles that leads to PL quenching of nanotubes with larger energy band gap compared to those with smaller band gap.

2. Experimental section

Poly(9,9-di-(N,N-dimethylaminopropylfluorenyl)-2,7-diyl) (PFDMA) was prepared via Pd(0)-catalyzed Suzuki coupling of dibromo- and diboronic acid pinacol ester- fluorene monomers. This polymer was quarternized by the addition of methyl halide to form PFAB. The detailed synthetic procedures and the characterization of the polymers are included in the [Supporting Information](#). The molecular weight of PFDMA was found to be almost one order of magnitude lower than that of PFO, which was purchased from Sigma-Aldrich (The Netherlands) and used as received.

Two kinds of carbon nanotubes, Comocat SWCNT (C-NT) from Southwest Nanotech and Hipco SWCNT (H-NT) from Carbon Nanotechnologies, were used as received. The Comocat SWCNTs were characterized by a narrower diameter distribution in respect to the Hipco SWCNT (H-NT). For the preparation of SWCNT dispersions, dry nanotubes were added to 6 ml of polymer solution in a weight ratio of 1 mg SWCNT to 1–10 mg polymer, and the mixture was sonicated for 4 h in a tabletop ultrasonic bath (VWR, The Netherlands). The weight ratio was kept fixed, because the selectivity of all the polymers remains constant when the weight ratio of SWCNTs to polymer is in the range between 1:1 and 1:10 (See [Supporting Information Fig. 3](#)).

After sonication, the crude dispersion was centrifuged at 5 k rpm for 20 min, and the aqueous dispersion was ultracentrifuged at 65 k rpm for 2 h. The supernatant was then removed for further measurements. Absorption spectra were recorded with a Perkin-Elmer UV-vis-near-infrared spectrophotometer (Lambda 900). Steady-state and time-resolved photoluminescence measurements were performed, exciting the solutions at 760 or 380 nm by a 150 fs pulsed Kerr mode locked Ti-sapphire laser. The steady-state PL of SWCNTs and polymers were measured with InGaAs and Si-CCD detectors, respectively. The time-resolved PL of the dispersion was recorded by a Hamamatsu streak camera working in synchroscan mode, with photocathode sensitive in the near infrared spectral range. All the measurements were performed at room temperature, and the spectra were calibrated for the instrumental response.

3. Results and discussion

The absorption and photoluminescence spectra of diluted solutions of PFAB, PFDMA, and PFO are shown in [Fig. 1](#). All

samples showed a broad absorption ranging from 350 to 400 nm with absorption maxima at 393 (PFAB), 379 (PFDMA), and 386 nm (PFO), respectively. In the case of PFO/toluene solution, the main peak with no vibronic structure is assigned to the α -phase, and the resolved shoulder at 436 nm is due to the β -phase, as reported in previously [16]. PFDMA has short N,N-dimethylamino side chains at the 9 position of the fluorene unit and shows a better solubility than PFO, which exhibits linear octyl chains. This is evidenced both by the faster dissolution and by the lack of shoulder in the absorption spectrum of PFDMA. The main absorption peak of PFAB is red-shifted by 14 nm compared to that of PFDMA. This spectral difference may be due to the charged side-chains or to the larger dielectric constant of the environment (D₂O = 78 vs toluene = 2.4). The steady-state PL spectra of all the samples display two sharp bands and a weak shoulder in the range of 400–500 nm ([Fig. 1](#)).

The absorption and photoluminescence spectra of the SWCNTs dispersed with the three polymers at the weight ratio of 1–5 are shown in [Fig. 2](#). Both absorption and PL spectra reveal a large difference in the capability of PFDMA and PFAB to disperse SWCNTs selectively in respect to PFO. It should be mentioned that not only the structures of the polymer but also the weight ratio of the polymer and SWCNTs might influence the selectivity for specific carbon nanotube species, as was recently reported [17]. However, in our study we found that the selectivity of the three polymers remains constant when the weight ratio of SWCNTs to polymer is in the range between 1:1 and 1:10. It is logical to conclude that the different selectivity is caused by the polymer structure and not by other effects.

In the absorption spectrum of the PFO sorted SWCNTs ([Fig. 2c](#)), the peaks at wavelength higher than 900 nm are the E₁₁ transitions of 5 different SWCNT species corresponding to (7,5), (7,6), (8,6), (8,7), and (9,7), according to the empirical kataura plot [18,19]. In the case of Comocat SWCNTs dispersed with PFDMA in toluene ([Supporting Information Fig. 1](#)), the absorption intensity of the (6,5) tubes increases, and new peaks at longer wavelengths, corresponding to larger diameter nanotubes, were observed.

PFAB proved to be effective for SWCNT dispersion in D₂O ([Fig. 2a](#)). However, in this case ultracentrifugation was necessary for the separation of the sorted SWCNTs and bigger bundles by buoyant forces.

This is understandable by simply taking into account the density of the two solvents: 1.1 g/cm³ for D₂O and 0.9 g/cm³ for toluene. The sonicated SWCNT dispersion with PFAB was ultra-centrifuged at 65 k rpm for 2 h, as was the SWCNTs dispersion in standard aqueous surfactants [4]. By using optical measurements on the dispersion, we found that PFAB in water shows similar selectivity for SWCNTs as PFDMA in toluene (See [Fig. 2a and b](#) and [Supporting Information Fig. 1](#)). Moreover, the selectivity shown by PFAB in D₂O was not dissimilar to that observed for SDBS-based dispersions.

The absorption spectra of SWCNT dispersions of PFAB and PFDMA ([Fig. 2a and b](#)) show higher background intensity and less-resolved features than the PFO-based SWCNT dispersion ([Fig. 2c](#)), indicating the presence of more residual bundles in solution. Moreover, optical transitions from metallic tubes in the range of 500–600 nm were observed

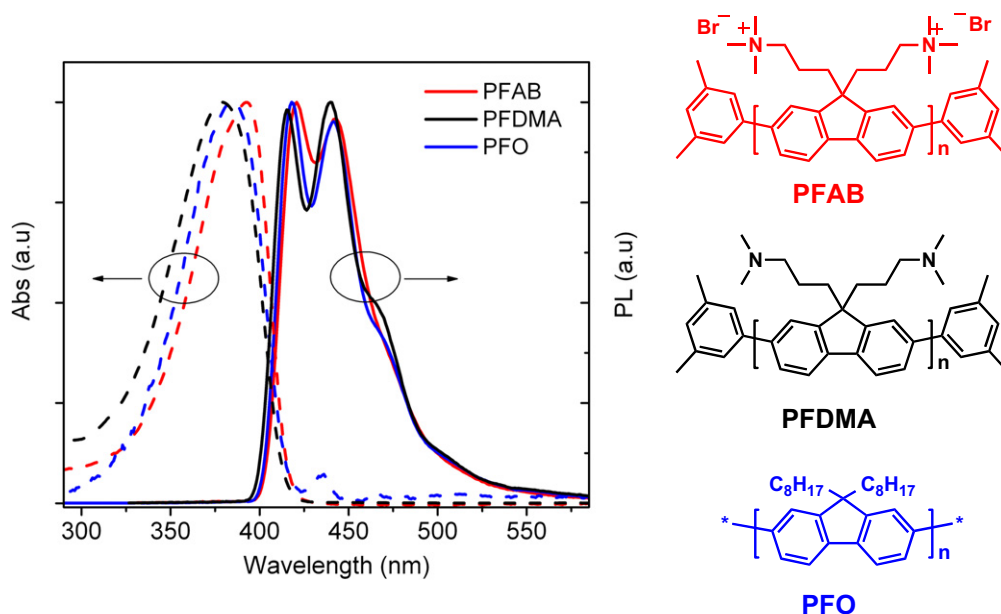


Fig. 1 – Absorption (dashed lines) and photoluminescence (solid lines) spectra of PFO (blue lines), PFDMA (black lines) toluene solutions and PFAB (red lines) D₂O solution. The spectra are normalized at their maximum intensity. The chemical structures of the polymers are shown at the right side of the figure.

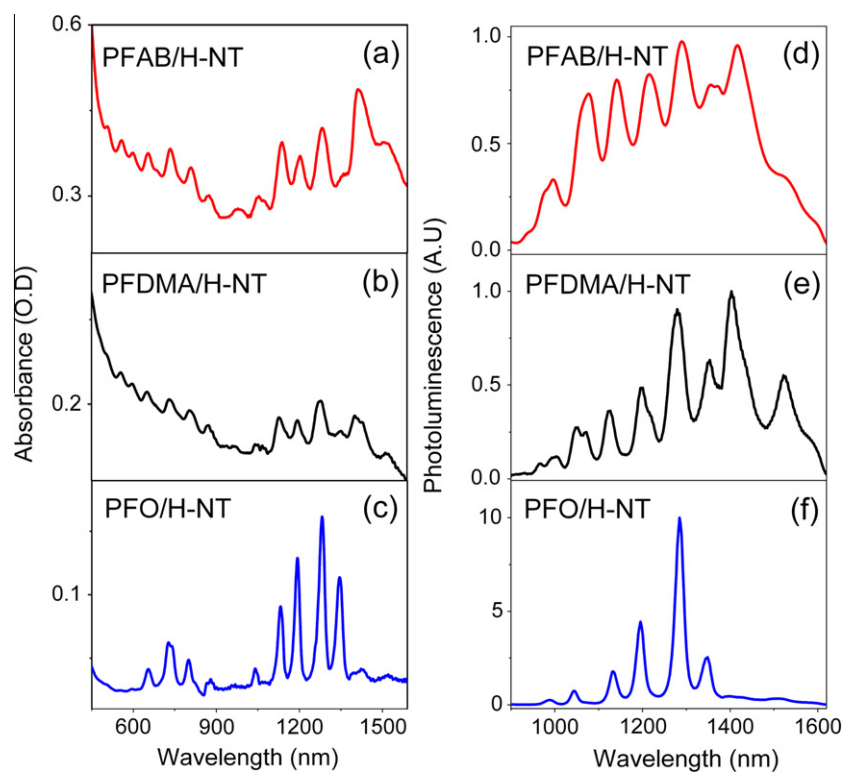


Fig. 2 – Absorption and photoluminescence spectra of Hipco SWCNTs dispersion with different polymers and solvents. Absorption spectra of SWCNTs dispersed with: (a) PFAB/D₂O; (b) PFDMA/tol; (c) PFO/tol. PL spectra of SWCNTs dispersed with: (d) PFAB/D₂O; (e) PFDMA/tol; (f) PFO/tol.

exclusively in the dispersions with PFAB and PFDMA (Fig. 2a and b). By taking into account the absorption cross section $\alpha(E_{11}^S) \approx 1 \times 10^{-17} \text{ cm}^2/\text{atom}$ for dispersed carbon nanotubes [20], we estimated their concentration to be about 0.5 $\mu\text{g/ml}$

for PFO/toluene, 0.8 $\mu\text{g/ml}$ for PFDMA/toluene, and 1.2 $\mu\text{g/ml}$ for PFAB/D₂O dispersion, respectively. In general, although the amino-substituted polymers yield higher total amounts of dispersed nanotubes, the selectivity of these polymers

for semiconducting carbon nanotubes is lower than that of PFO.

Steady-state photoluminescence measurements were used to probe individual SWCNTs and/or small semiconducting bundles in the three dispersions (Fig. 2d–f). The PL spectrum of the PFO dispersed SWCNTs, depicted in Fig. 2f, shows the highest PL intensity and the least full width at half maximum (FWHM) of the single peaks. Lower PL intensity and broader peaks were found for PFDMA/toluene and PFAB/D₂O dispersions (Fig. 2d and 2f). This is a further indication of the presence of bundles in the solution and is therefore in accordance with the results of the absorption measurements.

In the case of PFAB/D₂O dispersion, the PL spectra of the SWCNTs (Fig. 2d) are red-shifted by about 8 nm compared to those dispersed with PFDMA and PFO in toluene (Fig. 2e and f). Such a shift has been explained previously as due to the dielectric constant of the solvent [21]. In order to rule out any effects due to the counter ions present in the side-chain of PFAB, we studied the SWCNT dispersion in aqueous solution using a polymer with the same structure but with a different counter ion, namely poly(9,9-bis[3'-(N,N,N-trimethylammonium)-propyl]-2,7-fluorene di-(methyl sulfonate)) (PFAS). Optical experiments showed identical results for dispersions with this polymer as for PFAB-wrapped SWCNTs (See Supporting Information Fig. 2). This comparison demonstrates that the counter ions of PFAB do not affect the capability of the polymer to wrap around SWCNTs and leave the physical properties of the nanotubes unchanged.

It is important to note that Comocast SWCNTs sorting was also obtained and showed analogous behavior to the Hipco tubes (Supporting Information Fig. 1).

We conjecture that the different selectivity for SWCNTs of PFDMA and PFAB with respect to PFO is due to their different side-chain structures and molecular weights, which leads to dissimilar interactions of the polymers with the nanotube side-walls. It is necessary to stress that in addition to the difference in the chemical nature of the side chain, the molecular weight of PFDMA (and PFAB) synthesized via Pd(0)-catalyzed Suzuki coupling is much lower than that of PFO, which may also influence the interaction of the macromolecule with the carbon nanotube's walls. On the other hand, the well-known affinity of amino groups to carbon nanotubes could influence the recognition capability of the PFDMA as well [22]. Further studies that take these possibilities into account are currently underway.

The dynamics of the excitons in SWCNTs was studied with the aid of a high resolution streak camera (~3 ps), sensitive in the near infrared range. Fig. 3 shows the spectrally and time-resolved PL of the three dispersed SWCNT samples (Fig. 3a–c) and the PL decays in different spectral ranges corresponding to the emission of (6,5) and (7,5) tubes (Fig. 3d–f) compared with the time response of the instrument. The PL decays of both (6,5) and (7,5) tubes can be fitted with a bi-exponential function, where τ_1 and τ_2 represent the short and long time constants, respectively. The first decay component, τ_1 , is in the range of 4–14 ps and varies depending on the polymer

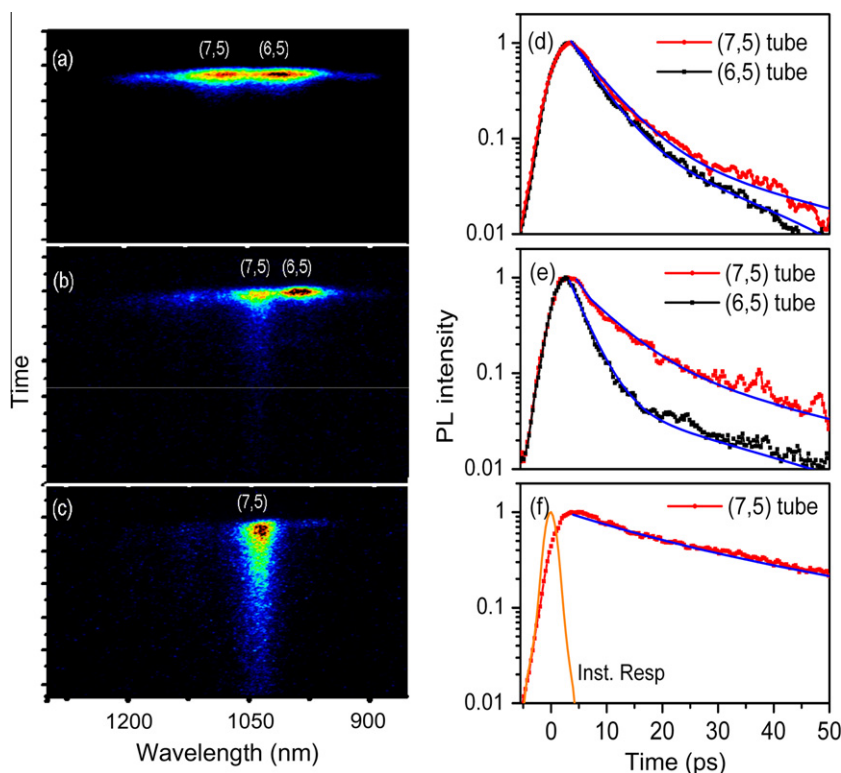


Fig. 3 – Intensity matrix of spectrally and time-resolved PL of nanotubes sorted with: (a) PFAB/D₂O; (b) PFDMA/tol; (c) PFO/tol. PL decays (6,5) and (7,5) SWCNTs dispersed with: (d) PFAB/D₂O; (e) PFDMA/tol; (f) PFO/tol. The blue lines are the fitting curves with two exponential components. The instrument response time is shown in orange. The intensity is normalized at $t = 0$. The samples were excited at 760 nm.

used for the dispersion. For the PFO/toluene system, the (7,5) tube has $\tau_1 = 14$ ps (Fig. 3f, Supporting Information Fig. 3), while the decay of the (6,5) tubes is not considered here, due to the negligible PL intensity. For PFAB-wrapped tubes, the first decay component of both tubes is of 5 ps and 6 ps, respectively (Fig. 3d), where τ_1 is 4 ps for (6,5) tubes and 8 ps for (7,5) tubes for PFDMA-based hybrids (Fig. 3e).

All the dispersions were excited at 760 nm, which is a non-resonant condition for (6,5) and (7,5) tubes. From this level the excitation relaxes to the excitonic state in sub-picosecond time scale. The first decay component in the range of 4–14 ps is attributed to the non-radiative loss of 'bright' exciton towards intrinsic 'dark' excitonic states, wall defects, and by inter-tube interaction in residual bundles [23–26]. Conversely to the first decay time, both nanotube species in all the dispersions show a long decay component τ_2 in the range of 28–40 ps, which we attribute to the intrinsic lifetime of this SWCNT species. This is consistent with previous reports regarding small diameter tubes [27]. It is important to note that the wrapping of the polymer around the SWCNTs modifies locally the dielectric constant; this is especially important when using media with dielectric constant very different from the media of the polymer (2–2.5).

The variation of the PL lifetime of the first decay component in the polymer dispersed carbon nanotubes is in agreement with an energy transfer between species of different band-gap, which appears to commonly occur in SWCNT bundles [28]. This interaction is one of the most elusive effects in carbon nanotubes photophysics, in that the investigation of these effects is strongly limited by the quality and control of the nanotubes separation [29]. Up to now, very few studies on energy transfer in SWCNT aggregates based on steady-state measurements such as high-resolution optical microscopy and photoluminescence excitation spectroscopy have been reported [30–35]. It is known, however, that precise information of the energy transfer can be obtained only with time-resolved spectroscopy [36].

Among all the species of nanotubes in the dispersions, (7,5) and (6,5) tubes have larger energy band gaps, which make their emission properties sensitive to inter-tube energy transfer when they are in bundles. As we have demonstrated above, SWCNT dispersions with PFO/toluene solution contain almost no SWCNT bundles. This is further evidenced by the fact that the decay time is the longest of all three dispersions ((7,5) tube ($\tau_1 = 14$ ps; $\tau_2 = 38$ ps)). The dispersions with PFDMA and with PFAB contain small bundles, and the PL decay of the (7,5) tube are $\tau_1 = 8$ ps, $\tau_2 = 35$ ps and $\tau_1 = 6$ ps, $\tau_2 = 30$ ps, respectively. Moreover, in the PFDMA/toluene dispersion the fluorescence decay of (6,5) tubes (larger band gap tube) is faster (4 ps) than that of (7,5) tubes (8 ps), suggesting an efficient interaction between the larger band-gap nanotube (6,5) and the surrounding smaller band-gap nanotubes ((7,5) and smaller band gap tubes).

Very recently, Lüer et al. [37] reported ultrafast (less than 10 fs) energy transfer in small SWCNT bundles by transient absorption spectroscopy. Although our measurements have inferior time resolution (3 ps), the results obtained are in agreement with this recent report.

Finally, our study demonstrates that time-resolved spectroscopy is the better tool in order to evaluate the quality

and the separation of semiconducting SWCNT dispersion because it allows for the observation of inter-tube energy transfer in small bundles.

4. Conclusion

We have presented the photophysics of semiconducting SWCNTs sorted by wrapping with different polyfluorene derivatives. The side chain functionalities were used for achieving SWCNT dispersion in different solvents. We demonstrated that SWCNT dispersions are influenced by the molecular weight and the structure of side-chains of the polymer. The intrinsic lifetime of the small diameter SWCNTs measured in ensemble was found to be in the range of 28–40 ps. Moreover, we provided evidence of energy transfer from tubes with larger band gap to those with smaller band gaps occurring in SWCNT bundles. These results confirm the results of poor selectivity found in the steady state experiments for the PFAB and PFDMA and further demonstrate that time-resolved PL is a very efficient analytical tool to estimate the degree of individual isolation of semiconducting SWCNTs in solution.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.carbon.2010.09.036](https://doi.org/10.1016/j.carbon.2010.09.036).

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